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STRESS CORROSION CRACKING OF HIGH STRENGTH STEELS

Contract DA-04-495-ORD-3069

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Structural Materials Division

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INVESTIGATION OF STRESS-CORROSION CRACKING OF HIGH-STRENGTH ALLOYS

Contract DA-04-495-ORD-3069

Written by:

R. B. Robinson
R. J. Uzdarwin

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Approved by:

R. F. Kimpel
Assistant Chief Engineer
Structural Materials Division

AEROJET-GENERAL CORPORATION

Azusa, California

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CONTRACT FULFILLMENT STATEMENT

This is the third in a series of quarterly progress reports submitted in partial fulfillment of the contract.

I. OBJECTIVES

The objectives of this program are

A. To study the susceptibility to stress-corrosion cracking of rocket-motor case materials: e.g., Vascojet 1000, Ladish D6AC, and Type 300M alloy steels, AM355 and PH 15-7 Mo stainless steels, and B120VCA titanium

B. To study the environmental parameters, including the atmosphere both outside and inside the rocket case, that affect the rate and extent of stress corrosion

C. To determine the effect of material parameters - composition, strength level, welding, microstructure, surface conditions, etc. - on the stress-corrosion process

D. To devise and evaluate techniques for preventing the stress-corrosion cracking of rocket-motor case materials.

II. SUMMARY

Environmental stress-corrosion studies were conducted on Vascojet 1000 alloy steel for 2 weeks. The only observed failures have been on the specimens heat-treated to approximately 240,000 psi, and in tap-water, distilled-water, and salt-water environments. No resolution of the discrepancies between the tensile stresses as calculated from tables and as measured by strain gages has yet been achieved.

The fabrication of specimens of the other five alloys (Ladish D6AC and Type 300M alloy steels, AM355 and PH 15-7 Mo stainless steels, and the B120VCA titanium alloy) is nearing completion. Environmental stress-corrosion testing of these specimens will soon be initiated.

III. WORK PROGRESS

A. INTRODUCTION

The stress set up within an elastic body is proportional to the strain to which the body is subjected by the applied load, according to Hooke's law, which may be rewritten simply as

$$\text{stress} = k \times \text{strain}$$

where k is a constant for the material of the elastic body. This constant is known as the modulus of elasticity of the material, and for most steel alloys is generally taken as 30×10^6 psi.

An elastic body may be loaded in three different ways. In one method the load tends to change the length of the body, in another to change the shape, and in the third to change the volume. Different procedures are necessary to evaluate the stress and strain for these cases, but Hooke's law applies equally to all three.

Flat beam specimens are used in the particular methods and techniques employed in studying the stress-corrosion rates for the alloys being investigated in this program. The amount of tensile stress induced in the specimen is directly dependent on the distance the beam is deflected. This deflection, in turn, depends on the length of the beam bent across a known distance. The stress can therefore be determined if both the length of the beam and the distance across which the beam is deflected are known.

Figures 1 and 2 show the apparatus used to obtain predetermined stresses. These stresses were actually determined from tables of tensile stresses in bent beams prepared by Dr. E. H. Phelps of the United States Steel Corporation.

In order to obtain a more accurate reading of the actual stresses obtained in the bent-beam specimens, Tatnall C6-141 foil-type strain gages, mounted longitudinally in the center of the top surface of selected specimens

(see Figure 3), were used to make a spot check. By taking strain-gage readings both before and after the specimens were stressed, the actual strain of the beam was obtained (in microinches). The maximum induced tensile stress was therefore computed by employing Hooke's law. However, the values obtained by the strain-gage method were considerably higher than those calculated from the tables (see Table 1). As a result, the environmental phase of the program was delayed until the problem was resolved. Dr. Phelps was consulted but required additional time for further consideration. In the meantime, the environmental stress-corrosion testing was resumed, because further delay in this phase of the program could not be tolerated without seriously hampering the overall progress.

B. VASCOJET 1000 STEEL

This is the only alloy to date for which environmental stress-corrosion testing has been initiated. Table 2 summarizes the test data. The only failures have been experienced with the specimens at the higher stress levels in the water and salt-water environments. However, sufficient data have not yet been accumulated to permit accurate comparisons and conclusions. Figures 4 and 5 illustrate the actual laboratory apparatus used in conducting the environmental stress-corrosion studies. Pyrex jars were used to permit periodic visual examination of the specimens without disturbing the testing. Continuous surveillance of the specimens was maintained for 16 hr each day, with careful inspections conducted every 4 hr.

Complete fracture of the specimen occurred in each failure to date. Some typical examples are illustrated in Figure 6. The failures did not occur in the center of the specimens, where the highest tensile stresses are believed to exist, but rather at some point away from the center. The reasons are not clear, and the matter is being investigated. Figure 7 illustrates a fracture edge, the tensile side being at the top. The cross section of a fractured surface is shown in Figures 8 and 9. This particular specimen was stressed to 180,000 psi and failed in a 3% sodium chloride solution after 6.7 days. Near the surface, where the failure had initiated, there are signs of intergranular corrosion. However, most of the fractured surface does not clearly define the

type of failure. Improved techniques are being investigated for better evaluation of the failed specimens.

C. TYPE 300M STEEL

Initial difficulties in the heat-treat response of this alloy were resolved after lengthy consultation with technical personnel of the Allegheny-Ludlum Steel Corporation, vendor of the material. A modified cycling that employed the double temper with the first draw 50°F below the final draw was recommended, and the results obtained were markedly improved (see Table 3). The test specimens were heat-treated according to the modified cycling, and are being prepared for environmental stress-corrosion testing by surface grinding and cutting to appropriate lengths.

D. LADISH D6AC STEEL

The mechanical properties of the test specimens prepared from this alloy are outlined in Table 3. The final operation of cutting these specimens to appropriate lengths is nearing completion, and environmental stress-corrosion testing is expected to begin shortly. Half of this material has been shipped to the Mellon Institute, as agreed, to permit the initiation of the Mellon program being conducted in conjunction with Aerojet's investigation.

E. AM355 STAINLESS STEEL

In evaluating the mechanical properties of this alloy, cold-rolled to various strength levels, some difficulties arose when the values obtained were not in agreement with those certified by the vendor. Technical personnel of the Wallingford Steel Company, vendor of the material, were consulted for a discussion of the results (see Table 4). Although several explanations were proposed, it was suggested that the best course of action would be for Wallingford to re-evaluate the material before any positive recommendations were made.

After the re-evaluation, Wallingford reported that the strength-level differences resulted from testing in different directions. Wallingford tested the material in the direction transverse to the rolling whereas the

Aerojet tests were in the longitudinal direction. The Mellon Institute likewise tested in the longitudinal direction, obtaining even higher values than Aerojet. To permit stress-corrosion tests of this alloy at lower strength levels, specimens in the direction transverse to the direction of the rolling are being prepared. The specimens previously fabricated in the longitudinal direction are being prepared for environmental stress-corrosion testing by cutting to appropriate lengths as calculated from the tables of tensile stresses in bent beams. The microstructure of this alloy is shown in Figure 10.

F. B120VCA TITANIUM

This alloy, cut into both transverse and longitudinal specimens, was aged in air at 900°F for varying periods to produce strength levels equivalent to those obtained in steel on a strength-to-weight ratio. Table 5 outlines the mechanical properties of this alloy. Because the aging was done in air, some slight oxidation of the surface occurred. The oxides were readily removed by pickling the specimens in a solution of the following composition:

	Wt%
Nitric acid	25 to 30
Hydrofluoric acid	3 to 4
Water	(Balance)

The maximum stock removal was 0.002 in. per surface. The cutting of the specimens was nearly completed and the environmental stress-corrosion testing of this alloy is expected to begin soon.

G. PH 15-7 Mo STAINLESS STEEL

This alloy was received from Wallingford cold-rolled to a 200,000-psi yield strength. It was determined that Wallingford's certified test data reflected the results of testing in the direction transverse to that of the rolling. Mechanical-test specimens for the evaluation of the aging of the alloy to yield strengths higher than 200,000 psi are being prepared. In

addition, environmental-test specimens are being fabricated so they will be ready for processing as soon as the preliminary evaluation is completed. The preliminary aging evaluation is expected to be finished shortly, whereupon both the transverse and longitudinal directions will be tested for mechanical properties.

H. TEST ENVIRONMENTS

The following environments are being used in studying the stress-corrosion rates for the materials being investigated in this program. The environments are representative of those that the materials undergo in the manufacture and testing of solid-rocket-motor cases.

1. Air
2. Distilled water
3. Tap water
4. High humidity
5. Salt water (3 wt% sodium chloride)
6. Inhibited water (0.25 wt% sodium dichromate)
7. Heat-treat salt solution (1 wt% Marquench salts)
8. Trichloroethylene
9. Cosmoline
10. Water-soluble oil
11. Water-soluble oil solution (4 vol% soluble oil)
12. Solid-propellant combination.

IV. FUTURE WORK

The environmental stress-corrosion testing of unwelded specimens of all six alloys is expected to be completed by the end of the next quarter, concluding the first year of the program.

The second year of work will include the evaluation of environmental stress corrosion of welded test specimens. In addition, protective systems for preventing stress corrosion in rocket-motor casings will be studied. The environmental stress-corrosion testing of both the first- and second-year phases of the program will be continued for extended periods on the most promising alloys or protective systems.

TABLE 1

STRAIN-GAGE EVALUATIONS*

Yield Strength 0.2% Offset psi x 10^{-3}	Maximum Tensile Strain, in. x 10^{6} (Strain-Gage Reading)	Maximum Tensile Stress, psi x 10^{-3} (Calculated from Strain-Gage Readings)	Maximum Tensile Stress, psi x 10^{-3} (Calculated from Tables)
194.0	7270	218.1	145.5
194.0	6360	190.8	145.5
212.0	6630	198.9	159.0
212.0	5770	173.1	159.0
237.7	7325	219.8	178.3
237.7	7330	219.9	178.3
242.4	8100	243.0	181.8
242.4	7490	224.7	181.8

*Tatnall C6-141 foil-type strain gages used.

TABLE 2

STRESS-CORROSION TEST DATA, VASCOJET 1000*

Yield Strength 0.2% Offset psi x 10^{-3}	Environment**	No. of Specimens	Time to Failure (days)	Total Testing Time (days)
194.0	1	3	NF***	17.0
	2	3		14.0
	3	3		14.0
	4	3		14.0
	5	3		14.0
	6	3		14.0
194.0	7	3	NF	14.0
212.0	1	3	NF	17.0
	2	3		14.0
	3	3		14.0
	4	3		14.0
	5	3		14.0
	6	3		14.0
212.0	7	3	NF	14.0
237.5	1	3	NF	14.0
	2	3		14.0
	3	3		14.0
	4	3		14.0
	5	2	NF	14.0
	5	1	13.7	--
	6	1	7.6	--
	6	1	7.8	--
	6	1	8.9	--
	7	1	6.9	--
	7	1	10.0	--
237.5	7	1	10.1	--

*Stressed at 75% of yield strength.

**Environments identified as follows:

1. Air
2. Marquench salt solution, 1 wt%
3. Sodium dichromate solution, 0.25 wt%
4. Trichloroethylene
5. Tap water
6. Distilled water
7. Sodium chloride solution, 3 wt%.

***No failure.

TABLE 2 (cont.)

Yield Strength 0.2% Offset psi x 10^{-3}	Environment	No. of Specimens	Time to Failure (days)	Total Testing Time (days)
240.0	1	3	NF	17.0
	2	3		14.0
	3	3		14.0
	4	3	NF	14.0
	5	1	2.7	--
	5	1	8.8	--
	5	1	9.7	--
	6	1	1.8	--
	6	1	3.2	--
	6	1	4.3	--
	7	1	1.2	--
	7	1	1.7	--
240.0	7	1	6.7	--

TABLE 3

MECHANICAL PROPERTIES OF LOW-ALLOY STEELS

Material	Yield Strength 0.2% Offset psi x 10^{-3}	Tensile Strength psi x 10^{-3}	Elongation (%) in 2 in.	Rockwell C Hardness
Vascojet 1000*	194.0	232.9	8.5	46.7
↓	211.9	254.8	8.0	50.2
	237.7	299.5	6.7	54.5
Vascojet 1000*	240.0	307.2	7.0	55.3
Ladish D6AC*	197.6	208.3	8.2	44.7
↓	222.7	235.4	6.3	47.7
	235.0	254.5	5.2	50.3
Ladish D6AC*	251.8	281.2	5.0	51.8
Type 300M**	209.3	254.6	7.5	51.5
Type 300M**	232.0	268.5	6.7	53.2
Type 300M**	244.4	282.4	4.7	54.8

*Average values for the stress-corrosion test specimens.

**Preliminary test data.

TABLE 4

MECHANICAL PROPERTIES OF AM355 STAINLESS STEEL*

Evaluator	Testing Direction	Thickness in.	Yield Strength 0.2% Offset psi x 10^{-3}	Tensile Strength psi x 10^{-3}	Elongation (%) in 2 in.	Rockwell C Hardness
Aerojet	Longitudinal	0.038	245.7	263.0	16.0	53.0
Mellon	Longitudinal		250.0	261.0	14.5	**
Wallingford	Longitudinal		250.6	258.9	14.0	**
Wallingford	Transverse	0.038	210.0	275.5	11.0	52.0
Aerojet	Longitudinal	0.036	249.8	265.8	14.2	54.2
Mellon	Longitudinal		275.0	265.0	15.3	**
Wallingford	Longitudinal		259.7	264.4	14.0	**
Wallingford	Transverse	0.036	226.5	275.5	11.0	52.0
Aerojet	Longitudinal	0.033	278.4	295.1	4.0	55.5
Mellon	Longitudinal		302.0	311.0	3.8	**
Wallingford	Longitudinal		298.0	303.0	3.5	**
Wallingford	Transverse	0.033	251.5	296.0	7.5	54.0

*This alloy was cold-rolled to the various strength levels.

**Not reported.

TABLE 5

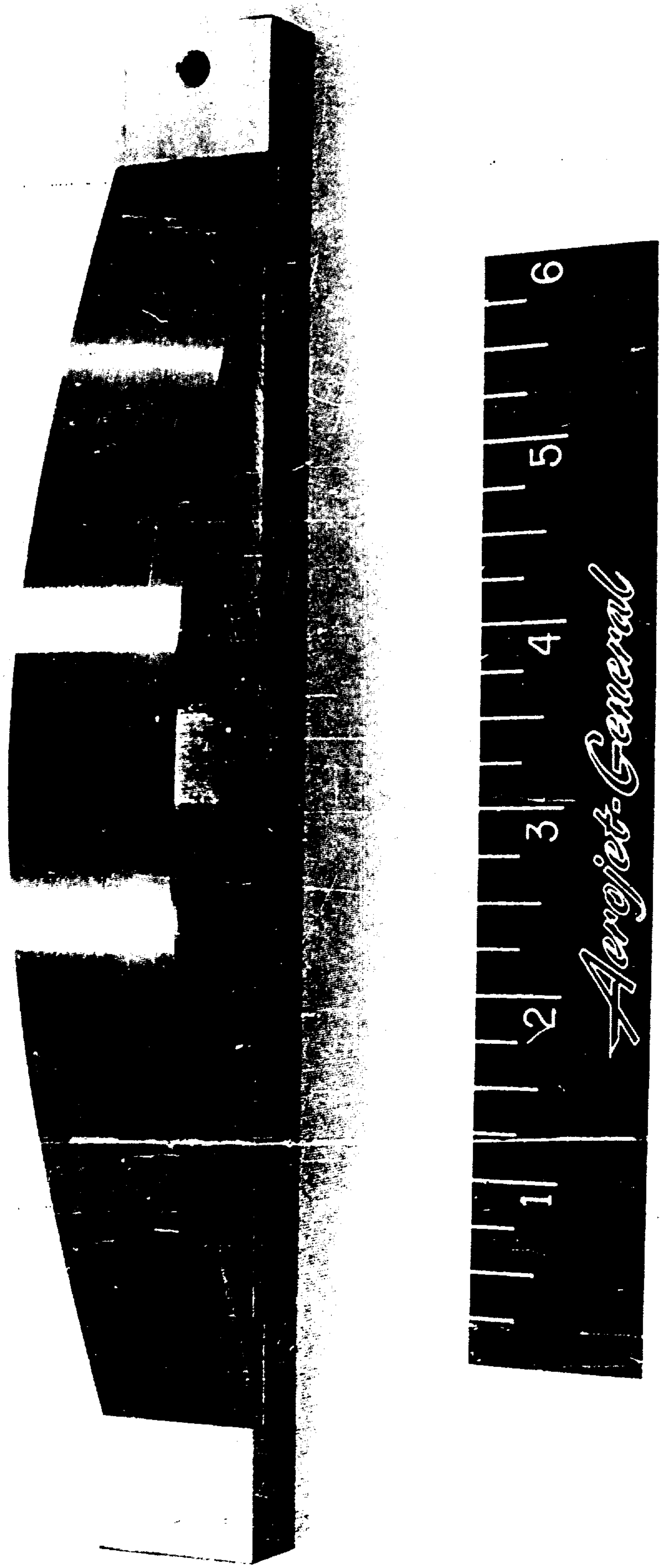
MECHANICAL PROPERTIES OF B120VCA TITANIUM ALLOY*

Aging Time hr	Testing Direction	Yield Strength 0.2% Offset psi x 10^{-3}	Tensile Strength psi x 10^{-3}	Elongation (%) in 2 in.	Rockwell C Hardness
0	Longitudinal	138.2	138.2	19.3	29.0
0	Transverse	135.7	138.6	19.3	28.7
12	Longitudinal	137.5	139.6	14.2	29.5
12	Transverse	139.6	141.8	12.8	30.0
45	Longitudinal	149.2	164.7	8.7	34.5
32	Transverse	145.6	155.7	11.5	32.5
75	Longitudinal	157.8	177.5	7.5	37.8
75	Transverse	166.1	186.8	6.0	38.8

*Average values for the stress-corrosion test specimens, aged in air for the times indicated.

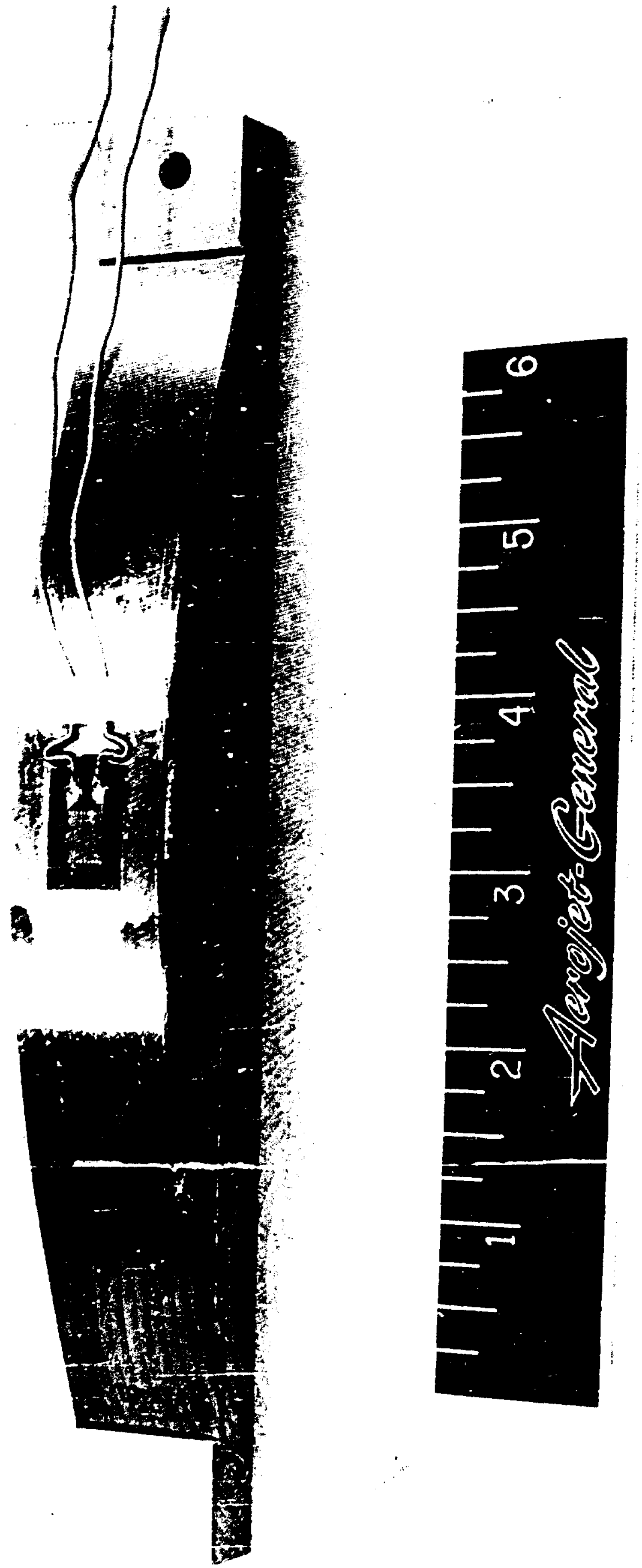
Test Specimen and Fixture Before Stressing

Figure 1



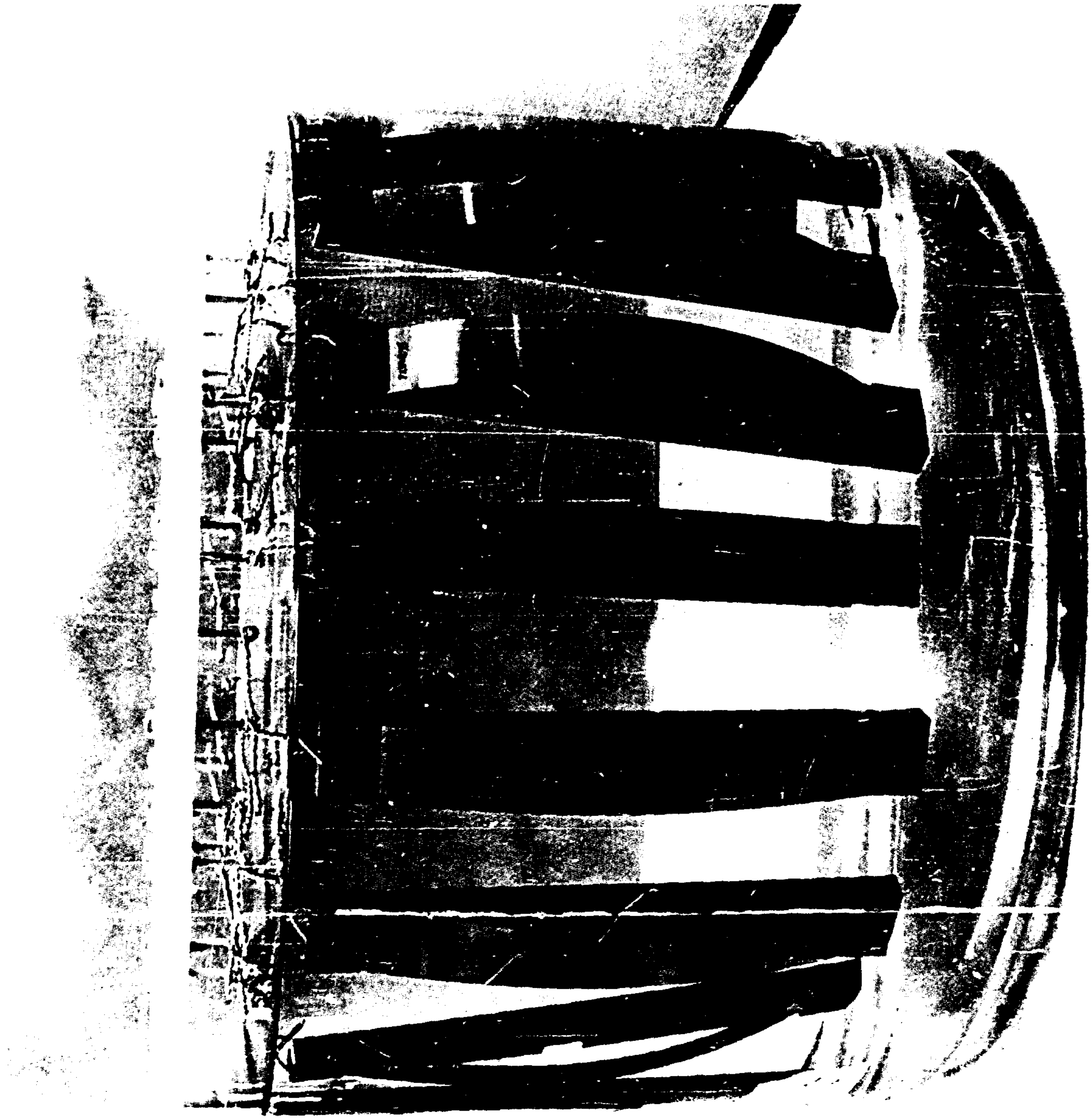
Test Specimen Stressed in Fixture

Figure 2



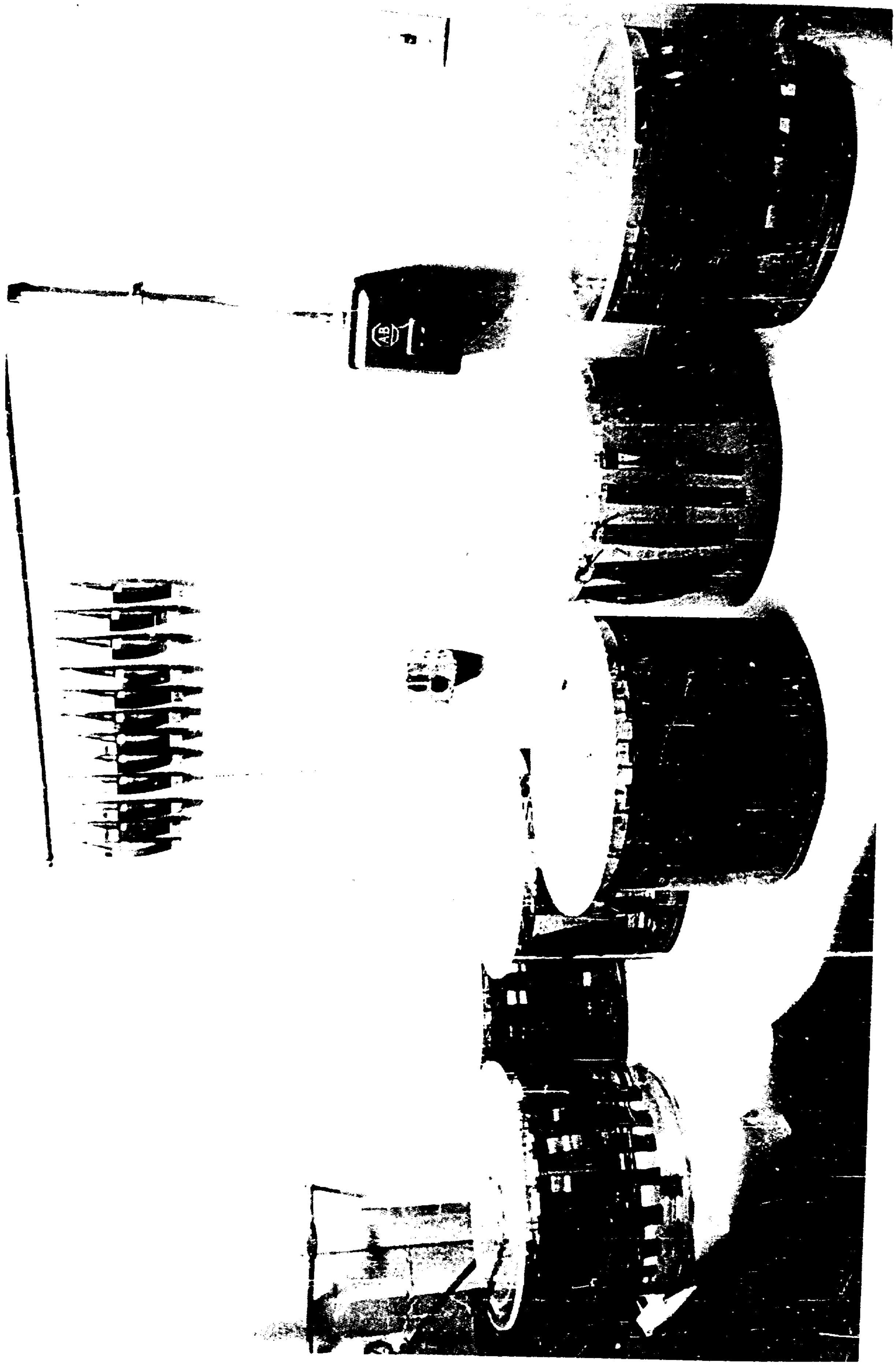
Test Specimen with Strain Gage Mounted,
Stressed in Fixture

Figure 3



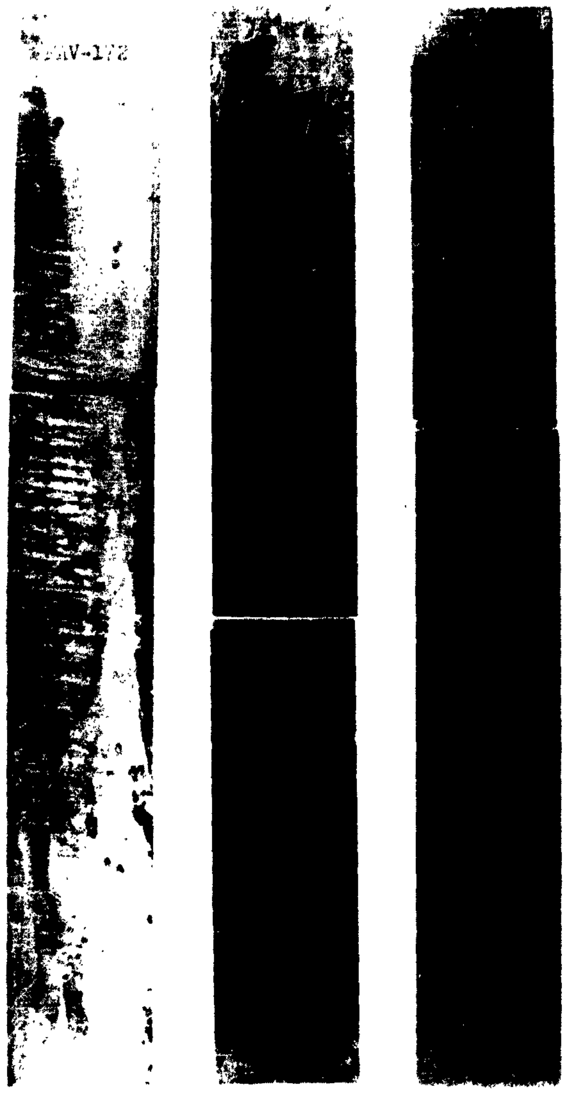
Specimens Undergoing Environmental Stress-Corrosion Testing

Figure 4



Environmental Stress-Corrosion Laboratory

Figure 5

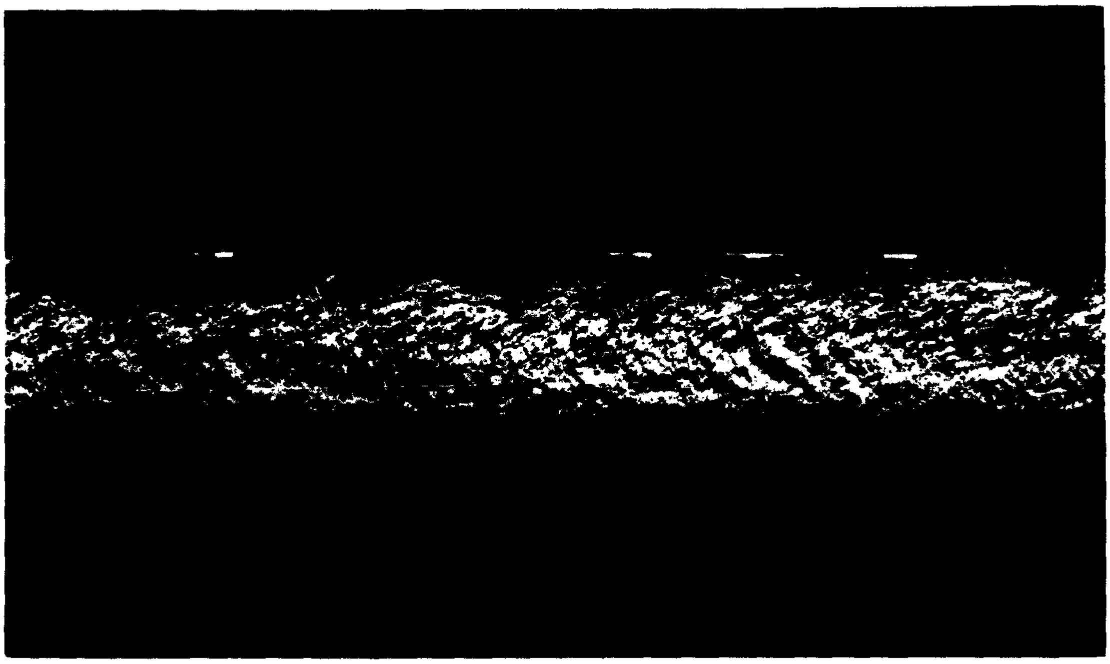


(A) (B) (C)

Vascojet 1000 Alloy Steel Specimens After Failure

(A) Tap Water, (B) Distilled Water, and (C) Salt Water

Figure 6

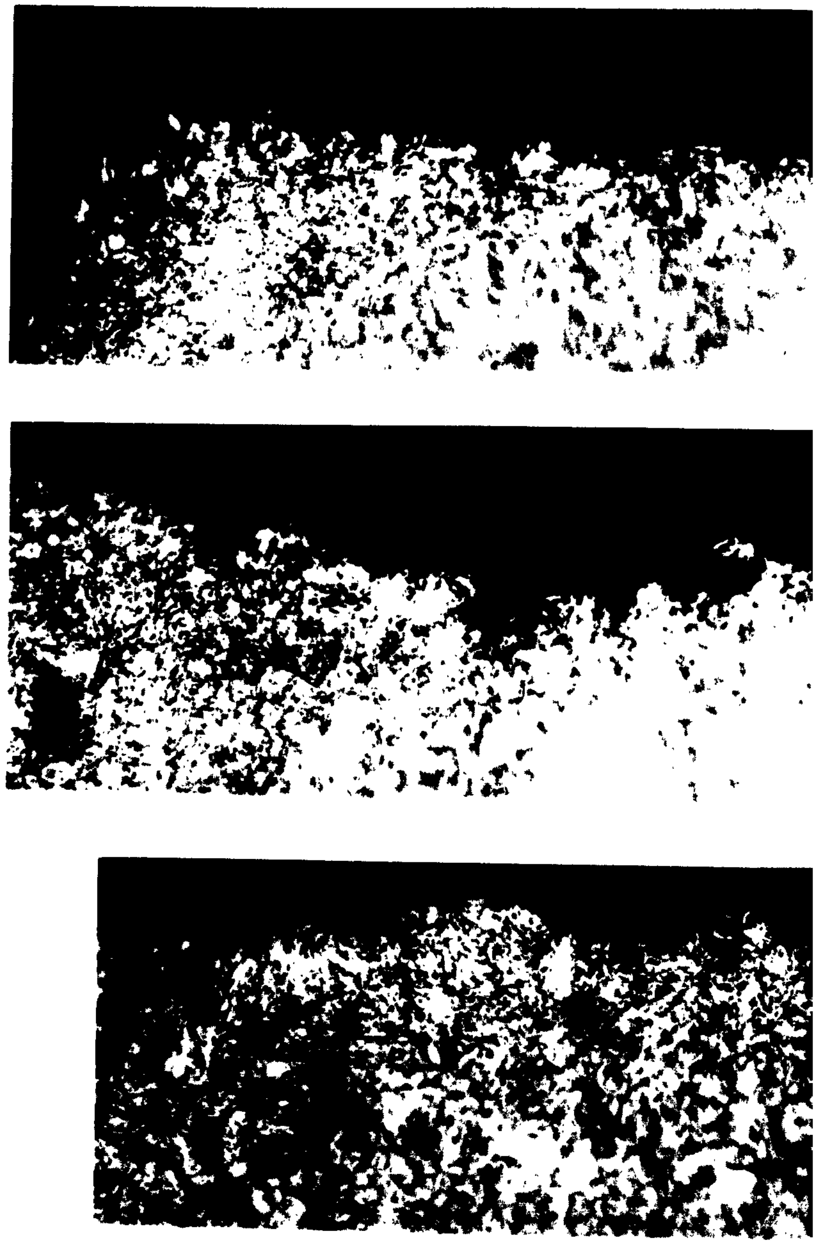


Fractured Surface of Failed Specimen
Mag. 15X

Figure 7

Cross Section of Fractured Surface
Mag. 75X Etch: Picric + HCl

Figure 8



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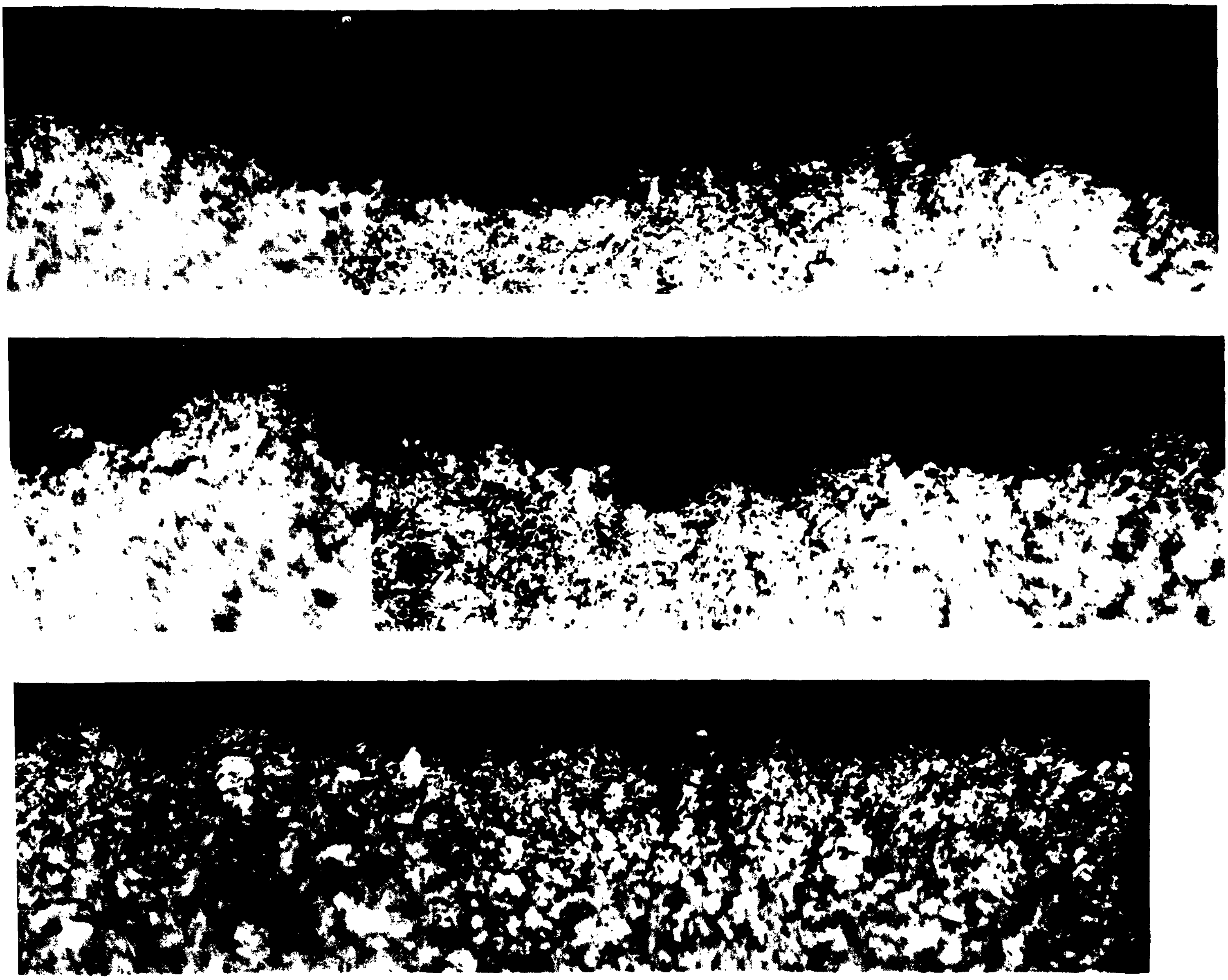
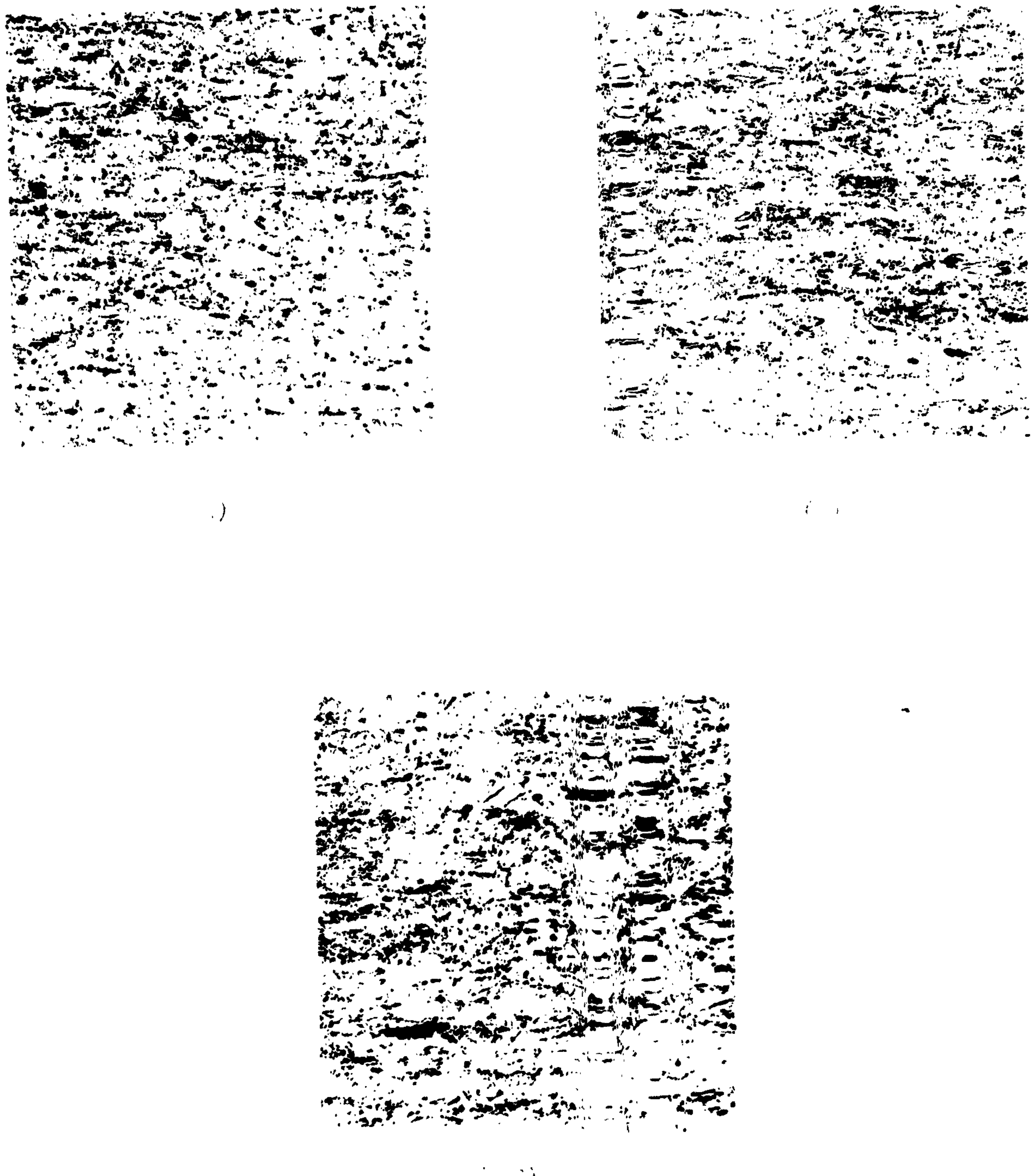
Microst



2

Microstructure of Cross Section of Fractured Surface
Mag. 500X Etch: Picric + HCl

Figure 9



General Structure of Cold-Rolled AM 355 Stainless Steel
Mag. 250X Etch: Ammonium Persulfate

(A) 0.033 in., (B) 0.036 in., (C) 0.038 in.

Figure 10